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Review on different methods for reducing outgassing rate from vacuum materials

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Abstract - *Outgassing plays an important role for the design and consideration of ultrahigh vacuum (UHV) system. Because they limit the lowest achievable pressure in a vacuum chamber, they considerably extend the time for high and ultrahigh vacuum to be reached and the outgassing molecules are a source of impurities in a vacuum chamber. So Reduction of the outgassing rates of the materials used in the construction of vacuum systems is essential if the UHV is to be achieved. In a metal system the residual gas is almost entirely hydrogen at UHV. So for reducing these effect of outgassing while making any vacuum related components, outgassing quantity of material should be as low as possible. There are lots of methods are available to reduce outgassing rate. Hence, it is proposed to understand outgassing effect in vacuum and to apply various techniques to vacuum components to reduce these effects.*

Keywords *– Outgassing, ultrahigh vacuum, vacuum chamber, coating, baking.*

I. INTRODUCTION

A. Outgassing

Outgassing is an evaluation of gas from solid or liquid in vacuum. Outgassing flux of a solid or liquid is quantity of gas leaving the surface per unit time at a specified time after the start of evacuation. [1]
Outgassing means usually two things:
1. Molecules diffusing through the bulk material of a vacuum chamber, entering the surface and desorbing from it.
2. Molecules which have been adsorbed previously, usually during venting of the vacuum chamber, that desorb again, when
the chamber is pumped to vacuum.

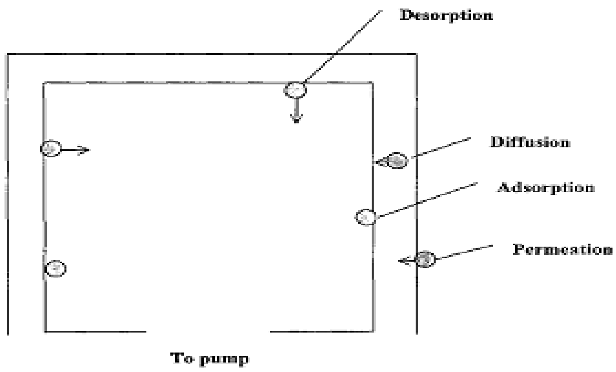

Figure 1. Sketch to show outgassing in vacuum with different ways

In any vacuum system that has reached equilibrium and in which leaks have been eliminated the pressure depends on the total outgassing of the system and the pumping speed of the pumps.

$$P = Q/S \quad \text{torr [1]}$$

Where Q is the outgassing rate in torr litre per second (torr 1. S^{-1}) and S is the pumping speed in l. S^{-1}. The rate of outgassing of a material is expressed in torr litre per second per squarecentimetre (torr l s^{-1} cm^{-2} or pa m s^{-1}) and the total outgassing of a system will be made up of the sum of the outgassing rate of each material present multiplied by its area.
A further source of outgassing is the vaporisation or sublimation of atoms or molecules from a material with a vapour pressure higher or comparable to the vacuum pressure to be applied to a chamber. Although this fact is trivial, it greatly limits the number of materials that can be used inhigh and ultrahigh vacuum chambers and can sometimes be a difficult problem to overcome. But the careful selection of materials will not be the subject of this paper and we restrict ourselves to the effects of desorption, out-diffusion and permeation of materials suitable for UHV applications.

B. Hydrogen in outgassing

Hydrogen is predominant residual gas at metal vacuum systems at very low pressure and the reduction in the hydrogen outgassing rate is most challenging problem in achieving vacuum of desired level [2].
Hydrogen outgassing can be well understood by ways of its outgassing. These includes a) the desorption/adsorption/diffusion equilibrium at the vacuum-metal interface, b)the effect of multiple energy stats of H atoms in the bulk metal, c) the physical adsorption of hydrogen on surfaces at cryogenic temperatures and d) the anomalous effects in pressure measurement of hydrogen with ionization gauges and residual gas analysers.

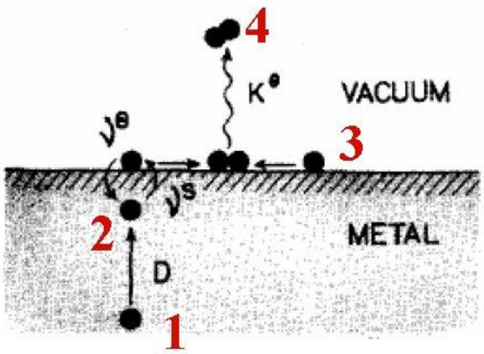

Figure 2. Schemating diagram of the elementary processes involved in the desorption of hydrogen from a metal [2].

C. Outgassing measuring methods
Following methods are used for measuring outgassing rate. An experimentalist must choose the most suitable method for particular application and obtaining all outgassing data.
Throughput method: In this method vacuum chamber is connected to apumping system through an orifice with conductance C. The pressure (p) in the chamber will be large relative to ultimate pressure of the system. For a given gas, net pumping speed will be independent of pressure and speed of pumps (in molecular flow region). Also it will be different for the various gas components in a gas mixture under molecular flow condition. The net pumping speed can be computed from conductance formula, calculated by Monte Carlo methods or view factor analysis, or measured.
Conductance modulation method: The conductance modulation method is a variant of the throughput method where the conductance of the pumping orifice is modulated.
Two path method: Another variant of throughput method is the two-path method. This method is especially suitable for the very low outgassing rate measurement. It also has the advantage that the x-ray limit and outgassing rate of the gauge are cancelled out by the difference method used. The outgassing rate of the test chamber is determined from the difference in outgassing rates, measured by the throughput method, when the gas flow is switched between two paths.
Gas accumulation method: The gas accumulation method is also known as the rate of rise method. When a test chamber is isolated from the pumps during the evacuation process, the pressure within the system will begin to decrease. The pressure will be minimum at instant of isolation and then it will gradually increases, till the outgassing rate equals to surface reabsorption rate. Usually at the timeof isolation, theoutgassing rate will be proportional to this initial instantaneousrate of pressure rise. Pumping down of the system may be continued by opening the valve to the pumps before the pressure has increased significantly [3]. This cycle is repeated at suitable intervals for obtain sufficient data to plot outgassing rate as a function of pump down time.
Mass loss measurements: The obtaining of outgassing data by mass-loss measurements is often referred as weight-loss measurements [3]. This method involves the measuring of the loss in mass of a material over specific time interval and at the recommended temperature. Continuous recording is preferred as the results give instantaneous rate measurements. A schematic diagram of themeasurement system for the mass loss method

II. LITERATURE REVIEW

For outgassing measurement system, measured quantity of outgassing should be as accurate as possible. This can be done by reducing outgassing quantity of samples with applying different techniques. According to these techniques there are many methods to reduce outgassing of samples. These methods are described in review.

D. Reduction in outgassing by coating

P. He et al [5]the stainless steel vacuum chambers of the 248m accumulator ring of Spallation Neutron Source (SNS) are coated with -100 nm of titanium nitride (TIN) to reduce the secondary electron yield. The coating is produced by D C magnetron sputtering using a 1ong cathode imbedded with permanent magnets.The required average operating pressure for the SNS storage ring is $5x10^{-4}$ Torr.The outgassing rates of several SNS half-cell chambers were measured with and without TiN coating, and before and after in-situ bake. One potential benefit of a TiN coating is to serve as hydrogen permeation barrier that reduces the ultimate outgassing rate. By varying the coating parameters, films of different surface roughness were produced and analysed by Auger electron spectroscopy, scanning electron microscopy and atomic force microscopy toillustrate the dependence of the outgassing on the film structure.

Table 1. Hydrogen Outgassing Rates for SNS Vacuum Chambers

Chamber	Q (torr-l/s cm^2)	Comments
1	2.5 * 10^{-13} (120 hrs., post bake)	in-situ 250°C bake, without TIN coating
2	2.1 * 10^{-13} (96 hrs., post bake)	in-situ 250°C bake, with high pressure TIN coating
3	1.9 * 10^{-13} (72 hrs., post bake)	in-situ 25O°C bake, with low pressure TIN coating

Yong Wang et al [6]TIN film was coated by DC sputtering on the inner face of a stainless steel vacuum chamber which is 86 mm in diameter and 2300 mm in length.The vacuum performances testing of the coated chamber has also been done, including thermal outgassing rate measurement, Photon stimulated Desorption (PSD) measurement, and Secondary Electron Yield (SEY) measurement of samples. Compared with those of uncoated stainless steel chamber, the results show that coating TIN film is a very effective method of the treatment of particle accelerator vacuum chamber.Outgassing rate is decreased with TIN coating in stainless steel as shown in figure 3. These difference is more with increasing the time. Also with time increasing outgassing rate is decreasing for both with coating and without coating.

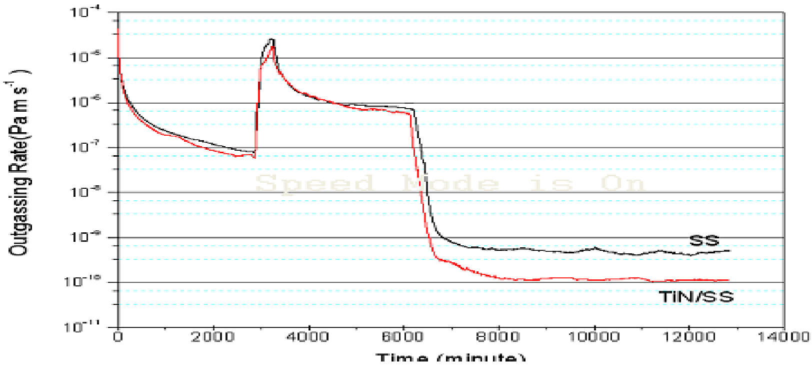

Figure 3. Compare of the outgassing rate for SS chamber and TIN coated SS chamber

F. Reduction of outgassing by baking

M. Bernardini et al [7]Hydrogen is most responsible gas of outgassing rates also in SS vacuum chamber. Heating the raw material at 400 °C in air was suggested as a money saving alternative to the classical vacuum heating at 950 °C.In this paper concluded that air bake-out drives out most of the hydrogen absorbed in the bulk stainless steel.
Results show that bake-out in air is effective in reducing the hydrogen outgassing rate of a very large stainless steel vacuum chamber. The hydrogen content and the diffusion parameters for a 304 L type stainless steel have been measured by desorption tests on small samples. It is concluded that the effect of the heating treatment in air is mainly to reduce the hydrogen content. Outgassing rate can be decreased with baking of materials as shown in figure 4.

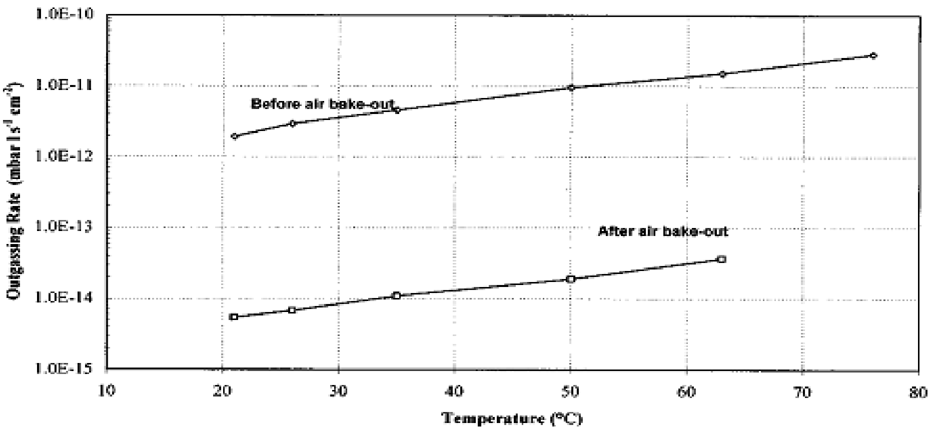

Figure 4. Hydrogen outgassing rate with and without air baking[7]

Y. Tito Sasaki et al[8] Significant reduction in the outgassing rate of 300-series stainless steel is routinely attained through combination of electropolishing and vacuum baking. Preferential removal of Ni, Fe, and Mn from the surface of stainless steel by electropolishing creates a chromium-enriched surface. It also reduces the atomic surface area of the work piece closer to its geometric surface area. When the material is vacuum fired to remove interstitial hydrogen, the resultant stainless steel exhibits an outgassing rate of about $2*10^{-15}$ torr l /cm^2 s, as well as drastically reduced adsorption, absorption, and catalytic behaviours.

Table 2. Hydrogen outgassing rates of differently prepared stainless steel

Stainless steal	**H_2 outgassing rate (torr l/s cm^2)**
Chemically cleaned	$6*10^{-13}$
Electropolished	$3*10^{-13}$
Vacuum fired	$1.3*10^{-13}$
Electropolished and Vacuum fired	$1.1*10^{-13}$

K Akaishi et al [9] In order to investigate the effect of bakeout on the dependence of outgassing rate on pumping speed in a vacuum system, the outgassing rates of a test chamber at room temperature without baking and after bakeout at 100 and 250°C were measured as a function of pumping speed. The measured outgassing rate q (Torr l/s cm^2) obeys power law

q = C(S/A)m where C and m (0 < m < 1) are constants, S/A is the pumping parameter defined as the ratio of the pumping speed S(l/s) and the surface area A(cm^2) of the chamber, and the variable range of S/A is from 2.46 x $10^{-5}$to 1.26 x 10^2 (l/s cm^2).With baking at 250°C the sticking probability of the wall is till kept high value in the region of pumping parameter less than the critical pumping parameter but with baking at 100° the sticking probability rapidly decreases in the region pumping parameter less than the critical pumping parameter. Pressure in vacuum system can be decreased with baking and also with increasing the baking temperature as shown in figure 5.

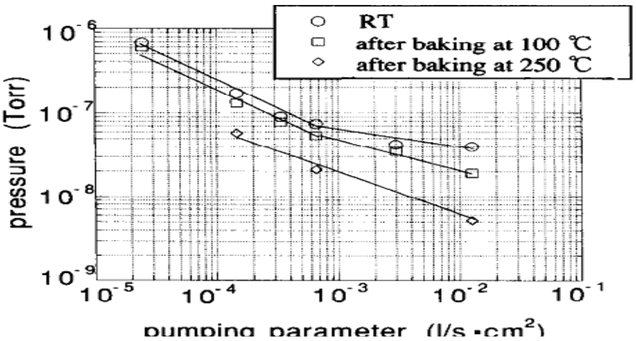

Figure 5. The measured pressures of the test chamber as a function of pumping parameter

G. Reduction by surface treatment

Arthur H. Tuthill et al [10] Passivation,, pickling, electro polishing, and in some circumstances, mechanical cleaning are the important treatments for successful performance of stainless steel used for piping , pressure vessel, tanks and machined parts in a wide variety of applications. The complete passivation treatment includes degreasing, immersion and rinsing; degreasing preferably in a non-chlorinated solvent, removes organic contaminants from the surface. Pickling removes heat tint and embedded contaminants. Several methods of evaluation of cleanliness are described in ASTM A 380.

D.N.Ruzic et al [11]Glow discharge cleaning removed the surface hydrocarbon layer,But deposited many constituents of the cathode and surrounding areas onto the sample.It also induced a high concentration of carbon which extended beyond the surface layer.

The extent of the carbon layer varied with theprior treatment of the surface. The presence of adsorbed gas in magnetic fusion devices is problematic; it degrades plasma Parameters. Discharge cleaning is often used to alleviate this problem.

F. Comparison of different materials

M.L.Stutzman et al [12]Outgassing measurements of three UHV materials (304 stainless steel (SS), 316L SS and 6061T-6 aluminium) were made using the conductance and accumulation techniques as given in the AVS recommended practice for outgassing measurements. Two independent techniques, called the orifice method and the rate of rise method, were used to measure the outgassing rates of each chamber. A description of the chambers, the measurement techniques and the result of the study are described in this paper. This paper also indicates outgassing rate of different materials. Outgassing rate reported here indicate that the three chamber materials outgassing rate independent of measuring technique.

Table 3. Different pressure for different materials

Material	Chamber pressure (torr)
304 stainless steel	$1.85 * 10^{-9}$
316L stainless steel	$2.46 * 10^{-9}$
6060-T6 aluminium	$1.96 * 10^{-9}$

III. CONCLUSION

After analysing above papers it is concluded that outgassing degrades the vacuum and increase the time to generate required vacuum level.There are many ways to measure outgassing rate and various methods are available to reduce outgassing rate from materials. These all techniques are used to reduce outgassing quantity up to some level. These techniques are coating, baking, surface treatments like electropolishing, rinsing, pickling, passivation etc. All techniques are important as single as well as combinations of them at a particular application. Combination of these techniques are more useful to reduce maximize outgassing rate. So, while choosing of materials and designing or manufacturing any vacuum vessel or chamber, consideration of outgassing effect is essential and have to be considered these techniques to reduce it. So, we can make vacuum components with less outgassing effect to achieve required level of vacuum within a certain time.

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